TREATMENT OF PRIMARY EFFLUENT[*]
BY LIME CLARIFICATION AND GRANULAR CARBON

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Introduction

Conventional systems for treating wastewater are usually composed of two processes: a primary treatment process which removes the bulk of the settleable solids from the wastewater by sedimentation, and a secondary treatment process which removes the bulk of the soluble organic matter by biological oxidation. These processes in combination have shown to be an effective and economical means of improving the discharge quality of wastewater. However, the processes are not without certain disadvantages.

First, the processes require considerable operating control and often generate operating problems of a complex nature. Second, they are easily upset and require time to regain efficient operation. And finally, they produce sludge at such a rate that it poses an ultimate sludge disposal problem of considerable magnitude.

Conceivably, each of these disadvantages could be largely eliminated if wastewater were treated by a system that utilized solely physical and chemical methods. Such a system is shown schematically in Figure 1.

In Figure 1, primary treatment is replaced by lime clarification with the added bonus of phosphate removal and secondary

treatment is replaced by activated carbon adsorption. Recalcination
of the spent lime and regeneration of the carbon make the system
essentially closed loop. The most intriguing aspect of the system
as a whole is that there are no large volumes of waste sludge to
contend with.

The results of applying activated carbon adsorption and
lime clarification techniques to wastewater renovation have been
widely reporter in the literature (1)(2)(3)(4)(5). However,
virtually all the work has been done on secondary effluent. The
number of reported instances in which lime clarification and carbon
adsorption have been applied to raw or primary wastewater are few.

Recently, a system utilizing lime clarification followed
by biological treatment was applied to raw wastewater (6). Results
showed that lime clarification with subsequent recalcination and
collateral combustion of the primary sludge solids was economically
competitive with conventional methods of removing suspended matter
from wastewater by sedimentation and ultimate disposal by incinera-
tion. The process had the added benefit of phosphate removal.

In another study, raw wastewater was lime clarified at
high pH and then carbon treated (7). The results presented were
interpreted as showing that the removal of soluble organic matter
by carbon is enhanced by the hydrolytic breakdown at high pH of
large molecular weight organic compounds into lower weight ones
which are more readily adsorbable. The results also give final
effluent COD values that compare favorably with those associated
with good secondary effluent.

Thus, to fully assess the feasibility of the system shown
in Figure 1, it will be necessary to answer two key questions.
First, can activated carbon adsorption compete with biological
oxidation as an economical method of removing soluble organics from
lime clarified raw wastewater, and second, can a complete physical
and chemical system of wastewater treatment produce water of a
quality at least equal to that produced by good conventional treat-
ment.

Work addressed to answering these questions is presently
underway at the Lebanon, Ohio pilot plant facility of the Federal
Water Pollution Control Administration. The purpose of this interim
project report is to give the preliminary results of the work.

Methods and Procedures

A flow diagram for the experimental system is shown in
Figure 2. Primary effluent from the Lebanon Municipal Treatment
Plant is fed to a lime clarification process for removal of suspended
matter and phosphates. A schematic of the lime clarification process
is given in Figure 3 and its operation is described in detail else-
where (1). Except for a brief operational period during which the

pH was greater than 11.5, lime clarification was carried out at a pH of 9.5. Following lime clarification and filtration through parallel dual-media filters, the clarified water is pumped through three carbon contactors for removal of soluble organic matter.

Each carbon contactor is 4 ft in diameter and contains 2400 lbs of 8x30 mesh granular activated carbon (a). This quantity of carbon fills the contactor to a depth of 8 ft. The contactors are operated downflow and in series.

Prior to dual-media filtration, the clarified effluent is adjusted to pH 8 with sulfuric acid to minimize calcium carbonate incrustation of the filter media. Following filtration, the clarified effluent is further adjusted to pH 7.5 with sulfuric acid prior to carbon treatment.

The lime clarification process is operated at a steady flow rate of 75 gpm. Part of this flow is stored for use in backwash of the dual-media filters, the remainder goes into a holding tank from which it is pumped at a constant rate of 48 gpm to the carbon contactors. During backwash of the dual-media filters, the water stored in the holding tank serves as feed to the carbon contactors.

At the flow rate of 48 gpm, contactor residence time based on an empty bed is 15 minutes each or 45 minutes total. This and other contactor constants are given in Figure 4.

Operation of the system is continuous over a 24-hour period and over run lengths of roughly one-month duration. During runs, the lime clarification process normally never requires shutdown; however, the carbon contactors require periodic shutdown which normally occurs when the headloss in the lead contactor is such that a flow of 48 gpm cannot be attained. When this occurs, the contactor is shut down, backwashed and then returned to service.

System performance was monitored initially by both grab sampling and composite sampling. Grab samples normally were taken manually every 3 hours at first, and later by automatic samplers every hour. Grab sampling was discontinued midway in the study and only composite sampling done.

Samples of the lime clarification process feed and product were taken for TOC, BOD, COD, turbidity, suspended solids, and phosphate determination. The feed and product of the carbon contactors were sampled for TOC, BOD, COD, and turbidity measurements. In addition, the product from carbon contactor one and two was sampled for TOC, BOD, and COD determinations. Late in the study, nitrogen forms around the system were determined. After establishing

(a) Filtrasorb 300, Calgon, Inc. Mention of specific proprietary equipment or products throughout this paper is for information purposes only and does not constitute endorsement by the Federal Water Pollution Control Administration and the U. S. Department of the Interior.

a ratio between TOC and BOD, and TOC and COD, only TOC was monitored around the system.

All samples were analyzed by Standard Methods (a).

Results and Discussion

Results will be presented in three parts: first those pertaining to carbon contactor performance; second, those pertaining to lime clarification at high pH; and last, those pertaining to the performance of the system as a whole.

Data on nitrogen and ratio results between TOC and BOD, and TOC and COD are given in the Appendix.

Carbon Contactor Performance

TOC profiles through the carbon contactors for increments of one million gallon throughput are given in Figure 5. The average TOC of the feed was 25 mg/l and the average product TOC was 11 mg/l. The range in feed TOC values was 20 to 33 mg/l. As can be noted, the effect of this variation in feed TOC on product TOC was damped by increased removal of TOC by contactor one. It is apparent that the lead contactor serves the important function of buffering increased TOC loadings.

In Table 1, the percent organic matter removed by the carbon contactors is given as TOC, BOD, and COD. As can be seen, organic removal was about the same regardless of the organic parameter utilized as a quantitative measure.

TABLE 1 - TOTAL PERCENT ORGANICS
REMOVED BY CONTACTORS

TOC[1]	57
BOD[2]	59
COD[2]	60

(1) Based on 5 MG
(2) Based on 3 MG

Organic removal as a function of contactor residence time is shown in Figure 6. The data suggest that the percent of the soluble organic matter removed cannot be substantially improved by increasing contactor residence time.

(a) Standard Methods for the Examination of Water and Wastewater 12th Edition, 1965, American Public Health Association, Inc. 1790 Broadway, New York, N. Y. 10019

Contactor loading data are given in Table 2. The loading in lbs TOC per lb of carbon shown for contactor one is considered very good since the contactor is only partially exhausted. In gallons of primary effluent treated, this loading in gallons treated per pound of carbon is roughly 2000. It appears realistic to project a treatment of 4000 gallons per pound of carbon (250 lb carbon/MG) before the carbon requires changing. This suggests carbon replacement costs of less than $0.07 per thousand gallons for one-time usage of the carbon and, if carbon regeneration is considered, the costs look economically attractive.

TABLE 2 - CARBON TOC LOADING - 5 MG

Contactor Number	Lbs TOC Removed	Lbs TOC Removed per Lb Carbon
1	367	0.153
2	169	0.070
3	51	0.021

TOC removed per lb of carbon as a function of TOC applied per lb of carbon is shown in Figure 7 for each contactor. These data represent the cumulative TOC applied and removed per million gallon increment of flow through the contactors. As can be seen, the TOC removal rate by contactor one decreased as the carbon became loaded with TOC. However, the TOC removal rate by contactor two increased as the carbon became loaded with TOC. This suggests that as contactor one becomes progressively exhausted it progressively passes an increasing amount of adsorbable organics to contactor two. TOC removal by contactor three was essentially constant at all loading rates. This seems to indicate that contactor two passes a relatively constant amount of adsorbable TOC to the third contactor.

Carbon Contactor Operational Problems

The carbon contactors became anaerobic early in the operational period. This was evident by the odor of hydrogen sulfide gas in the contactor effluent. The amount of hydrogen sulfide present was not measured quantitatively. An attempt was made midway in the study to operate the contactors in an aerobic condition. The first method used was to aerate the holding tank prior to the carbon contactors. Air was used first and then oxygen. Neither method injected enough oxygen into the feed to maintain the lead carbon contactor aerobic. Oxygen was then injected directly into the feed stream at the inlet to the lead contactor. This eliminated the hydrogen sulfide from the contactor effluent after a short operational period which indicated the contactors were operating aerobically. After a period of less than a day running time, however, a large leadloss developed in the lead contactor and maintaining the desired

flow rate through the contactor became impossible. The contactors were shut down, the lead contactor backwashed, and then all contactors put back into operation.

Rapid buildup of headloss across the lead contactor continued despite frequent backwash. The lead contactor was again shut down, partially drained, and its surface layer inspected. The layer was found to be greatly impregnated with calcium carbonate. The holding tank prior to the contactor was then drained and inspected and a heavy deposit of calcium carbonate was found at the bottom. This deposit probably accumulated during the high pH run when large quantities of lime were used to raise the pH above 11.5 in the clarifier. During backwash of one of the dual-media filters, the volume of water entering the holding tank is decreased by a half and the holding tank level drops to half or less. The momentum of the incoming water is probably sufficient enough at low water levels to create a bottom turbulence in the tank and the turbulence puts bottom deposits such as calcium carbonate into the feed to the contactor.

The holding tank was thoroughly flushed out with water, the lead contactor acid rinsed with 40 gallons of pH 2.0 sulfuric acid solution and backwashed, and then the contactor was returned to service. At this point the contactor throughput was approximately 1.5 million gallons. Headloss across the contactor was then normal. It was decided to cease feeding oxygen to the contactors and thereby let them become anaerobic. It was felt that the oxygen was promoting the growth of aerobic slime-producing microorganisms and contributing to leadloss buildup.

Following the acid rinse of the lead contactor and the cessation of oxygenation of the contactors, headloss buildup assumed a reasonable magnitude.

Lime Clarification at pH >11.5

As previously mentioned, it has been reported that lime clarification of raw wastewater at a high pH results in the hydrolytic breakdown of organic matter which subsequently enhances its removal by carbon adsorption (7). In an effort to evaluate this, the lime clarifier was operated at a pH greater than 11.5 over an eleven day operational period. The lime clarifier pH range for the period was 11.10 to 11.75 and the pH averaged 11.59. To achieve this pH level, lime dosages in excess of 1100 mg/l were necessary as compared to 200 to 300 mg/l to achieve pH 9.5. All other operational procedures and methods as previously described were the same. Operational sequence of the high pH run relative to the low pH runs was: 31 days at pH 9.5, 11 days at pH greater than 11.5, and 30 days at pH 9.5.

Removal of organic matter from primary effluent clarified at a pH greater than 11.5 is shown in Table 3. For comparative purposes similar data are given for the pH 9.5 runs. The high pH results were averaged over a throughput of 0.7 million gallons while

the low pH results were averaged over a throughput of 5.0 million gallons. Since a low pH run preceded and followed the high pH run, it is reasonable to assume that prior history of the carbon contactors did not prejudice the results to any significant extent.

As can be seen in Table 3, the pH level did not have a significant effect on product quality as measured by BOD, COD, and TOC, nor did it affect a greater percentage removal of organic matter.

TABLE 3 - CARBON ADSORPTION OF ORGANICS FROM
PRIMARY EFFLUENT CLARIFIED AT pH 9.5 and pH >11.5

	TOC	BOD	COD
pH 9.5: % Removed	56.0	58.7	60.4
Product Average, mg/l	11.0	10.0	31.0
Product Range, mg/l	2.9-22	2.0-23	11-70.6
pH >11.5% Removed	53	56.2	59.6
Product Average, mg/l	9.8	14.5	31.0
Product Range, mg/l	6.4-12.2	6.6-19.4	12.0-42.6

Some improvement in the turbidity of the effluent from the lime clarifier was noted at the higher pH. This was probably because of the removal of fine turbidity matter by the magnesium hydroxide floc which forms at a pH above 11.

Overall System Performance

Organic matter removed by the complete system as determined by TOC, BOD, and COD measurements is given in Table 4. As shown, lime clarification removed 76 percent of the organic matter. This suggests that the amount of organic matter present in Lebanon primary effluent that is amendable to removal by clarification and adsorption mechanisms is, on the average, 76 percent suspended and 24 percent soluble in form.

TABLE 4 - TOTAL ORGANIC REMOVAL FROM PRIMARY EFFLUENT
BY LIME CLARIFICATION AND CARBON TREATMENT

	TOC[1]	BOD[2]	COD[2]
Total Organic Removal			
mg/l	66	66	165
lbs/MG	550	550	1376
Percent Removed by Lime Clarification	76	76	76
Percent Removed by Carbon Adsorption	24	24	24

(1) Based on 5 MG
(2) Based on 3 MG

The percent removal distribution of the applied organic loading between the processes and that remaining in the product is shown in Table 5. As can be noted, 66 percent of the applied organic matter was removed by clarification, 21 percent by adsorption, and 13 percent was not removed by either process and remained in the product water. Assuming that essentially no soluble organic matter is removed by lime clarification, the data indicates that roughly 34 percent of the applied organic loading was soluble in form and, surprisingly, 13 percent of the soluble organics were not removed by carbon adsorption.

TABLE 5 - THE DISTRIBUTION OF THE APPLIED ORGANIC LOADING

	TOC[1]	BOD[2]	COD[2]
Removed by Lime Clarification, %	66	66	65
Removed by Carbon Adsorption, %	21	21	21
Remaining in Product, %	13	13	14

(1) Based on 5 MG
(2) Based on 3 MG

NOTE: Overall removal by lime clarification and carbon adsorption was 87% TOC, 87% BOD and 86% COD based on primary effluent. If 35% removal of BOD in the primary tanks is assumed, the overall BOD removal by primary treatment, lime clarification and carbon adsorption is 91.5%.

It is anticipated that raw wastewater could be fed directly to the lime clarifier (without primary treatment) without affecting product quality.

Turbidity, suspended solids, and phosphate removal data for the system are given in Figure 8. Removal in each instance was good. As would be expected, the carbon contactors tend to remove suspended matter and thus turbidity, that escapes the dual-media filters. Phosphates are not removed by activated carbon.

Organic concentrations of the feed and product stream as shown in Figure 8 indicate that overall system performance was quite good. The quality of the water obtained solely by lime clarification of primary effluent is probably equal to that discharged by many conventional treatment plants today plus the added benefit of phosphate removal. Final product water quality is equal to that required for direct discharge and for many reuse purposes.

In Table 6, a comparison is made between wastewater quality attainable by good conventional treatment and that attained in the present study by lime clarification and carbon adsorption. The data given for activated sludge treatment was obtained from measurements made on secondary effluent from a pilot plant activated sludge

unit operating at a constant feed rate. This unit utilizes primary effluent from the Municipal Treatment Plant and treats it by conventional activated sludge methods. The secondary effluent from this unit is normally of good quality with an organic carbon content of 20 mg/l or less and a turbidity of 10 or less JTU. As shown in Table 6, the quality of effluent produced by lime clarification and carbon adsorption was slightly better than that of the secondary effluent used as the standard, and probably significantly better in quality than the effluent produced by the majority of treatment plants now in operation that utilize conventional treatment methods.

TABLE 6 - COMPARISON OF CONVENTIONAL TREATMENT
AND PHYSICAL AND CHEMICAL TREATMENT

Treatment	BOD[1]	TOC[1]	JTU
Activated Sludge	79-13 [a]	59-12 [b]	2.4
		72-12	
Lime and Carbon	76-10	76-10	1.1

(1) Based on 1 MG
(a) First group of figures are influent, second group effluent
(b) Results from two separate sampling periods.

General Comments

        In assaying the results presented, two factors must be considered. First, the system under study was operated at a steady flow. The effect of diurnal variation in the flow of wastewater on the performance of the system was not determined. However, there is some evidence that several magnitudes of change in flow rate through a carbon contactor do not materially affect product quality (9)(10). The effect of variation in flow on the performance of the lime clarification process is not known.

        Second, the primary effluent used as the feed to the system is highly domestic in nature and is weak to moderate in strength. Conceivably the results would be significantly altered if a strong strength wastewater were used as the feed.

        The results must also be considered in light of certain deficiencies in the system. For one, the carbon contactors were not designed for efficient backwash. This most likely resulted in the establishment of a microbial population in the contactors, particularly the first one. Undoubtedly, some of the organic matter removed in the contactors was removed through biological metabolic activity. This poses the question as to whether or not the organic matter that survives carbon treatment is qualitatively and quantitatively the same whether or not the contactors are operated anaerobically or aerobically. It probably is not since the metabolic pathways are different and result in different end products. Possibly then, the magnitude of the

organic content in carbon created effluent could be influenced by operating conditions imposed on the contactors.

Second, operation of the contactors anaerobically is probably not desirable because of the ultimate problem of removing the hydrogen sulfide gas in the effluent. Air stripping would create odor problems. Oxidation to sulfate with chlorine or ozone could be costly. Costs involved, however, could be compared to the costs of oxygenating the contactors.

Summary and Conclusions

The results of the work thus far show that the treatment of primary effluent by lime clarification and activated carbon produces an effluent of good quality suitable for discharge. Averaged over a five million gallon throughput, effluent TOC and BOD were 10 mg/l with an overall range of 2-23 mg/l. Effluent turbidity averaged less than 2 Jackson turbidity units. Phosphate removals were consistently 90 percent or better. These characteristics are consistent with those associated with good quality secondary effluent plus the added bonus of phosphate removal.

Carbon contactor performance to date suggests that the lead contactor will treat 4000 gallons of lime clarified primary effluent per pound of carbon before requiring replacement. This projects a carbon make-up cost of $0.07/1000 gallons without recovery of carbon, and, possibly, less than $0.01/1000 gallons if the carbon is regenerated. These costs suggest that carbon treatment may be competitive with biological treatment as a means of removing soluble organics from primary or raw wastewater. However, sufficient information is not presently available to positively conclude this.

The results presented must be considered in the light of certain factors. First, the system was run at steady flow. Second, the feed to the system was a weak to moderate strength domestic wastewater. How the system would respond to a number of magnitudes of change in these factors is presently only speculative.

It is concluded that: (1) wastewater treatment to a quality level comparable to that achieved by good conventional methods is technically feasible using lime clarification and activated carbon treatment, and (2) projected costs based on carbon contactor performance thus far suggest that the removal of soluble organics from raw wastewater by activated carbon will be economically attractive.

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APPENDIX

Nitrogen Data

Some nitrogen data were gathered during the latter part of the study to determine the magnitude of the nitrogen forms present in lime clarified and carbon treated primary effluent. These data are given in Table 7. As can be seen, there was a substantial increase in ammonia nitrogen during carbon treatment. This is probably attributable to the ammonia produced by the hydrolysis of cellular matter present in the suspended organic matter reaching the carbon contactors. This is evidenced by the decrease in organic nitrogen across the carbon contactors. Nitrite and nitrate content of the final product was always less than 0.2 mg/l.

The amount of ammonia nitrogen present is typical of that found in most good quality secondary effluents since few conventional activated sludge treatment plants nitrify. Probably the organic nitrogen is somewhat less than that found in conventional secondary effluent.

TABLE 7 - NITROGEN BALANCES AROUND SYSTEM[1]

	NH_3-N	Org-N
Primary Effluent	11.5	4.2
Lime Clarified Primary	13.2	2.1
Lime Clarified-Carbon Treated Primary	16.8	1.0

(1) Based on 2 MG. All data in mg/l.

TABLE 8 - ORGANIC PARAMETER RATIOS

BOD/TOC Ratio

Lime Clarified Primary $\frac{25.8}{22.0} = 1.17$ $\frac{26.5}{23.2} = 1.14$

Carbon Contactor
No. 1 Product $\frac{15.5}{15.5} = 1.00$ $\frac{12.7}{11.2} = 1.13$

Carbon Contactor
No. 2 Product $\frac{11.4}{10.9} - 1.05$ $\frac{11.0}{9.6} = 1.15$

Carbon Contactor
No. 3 Product $\frac{11.9}{11.6} = 1.03$ $\frac{9.0}{9.6} = 0.94$

Average of all ratio results = $1.08 = \frac{BOD}{TOC}$

COD/TOC Ratio

Lime Clarified Primary $\frac{79}{22} = 3.59$ $\frac{72.0}{23.2} = 3.10$

Carbon Contactor
No. 1 Product $\frac{37.3}{15.5} = 2.4$ $\frac{40.2}{11.2} = 3.57$

Carbon Contactor
No. 2 Product $\frac{27.7}{11.6} = 2.39$ $\frac{36.4}{9.6} = 3.79$

Carbon Contactor
No. 3 Product $\frac{27.4}{10.9} = 2.51$ $\frac{35.5}{9.6} = 3.68$

Average of all ratio results = $3.13 = \frac{COD}{TOC}$

TABLE 9 - TOC DATA PER MILLION GALLON
INCREMENT OF THROUGHPUT

Gallons Throughput	Feed TOC, mg/l	Contactor Product TOC, mg/l		
		1	2	3
1035000	25.8	13.1	11.3	10.6
2005000	21.3	14.0	11.6	10.8
3040000	18.7	12.9	9.1	8.4
4058000	24.3	19.6	14.8	13.4
5024000	32.9	19.6	11.8	9.6
Average	24.6	15.8	11.7	10.6

TABLE 10 - LBS TOC APPLIED, REMOVED, AND REMAINING
PER MILLION GALLON OF THROUGHPUT

	LBS TOC APPLIED PER MILLION GALLON INCREMENTS OF THROUGHPUT					
	1	2	3	4	5	Total
Contactor 1	223	173	161	206	265	1028
Contactor 2	113	113	111	166	158	661
Contactor 3	98	94	79	126	95	492

	LBS TOC REMOVED PER MILLION GALLON INCREMENTS OF THROUGHPUT					
	1	2	3	4	5	Total
Contactor 1	110	60	50	40	107	367
Contactor 2	15	19	32	40	63	169
Contactor 3	7	7	7	12	18	51

	LBS TOC REMAINING PER MILLION GALLON INCREMENTS OF THROUGHPUT					
	1	2	3	4	5	Total
Product	91	87	72	114	77	441

$$\text{TOC Removed, \%} = \frac{\text{Total Removed}}{\text{Total Applied}} - \frac{587}{441 + 587} = 57$$

TABLE 11 - LBS TOC APPLIED AND REMOVED PER LB CARBON
PER MILLION GALLON INCREMENT OF THROUGHPUT

	LBS TOC APPLIED PER LB OF CARBON FOR MILLION GALLON INCREMENTS OF THROUGHPUT					
	1	2	3	4	5	Total
Contactor 1	.043	.072	.067	.086	.110	.430
Contactor 2	.047	.047	.046	.069	.066	.275
Contactor 3	.041	.039	.033	.053	.040	.205

	LBS TOC REMOVED PER LB OF CARBON FOR MILLION GALLON INCREMENTS OF THROUGHPUT					
	1	2	3	4	5	Total
Contactor 1	.046	.025	.021	.017	.045	.153
Contactor 2	.006	.008	.013	.017	.026	.070
Contactor 3	.0029	.0029	.0029	.005	.008	.021